# HYDRAULIC LIME – POZZOLANS: PROPERTIES, USES AND RESEARCH NEEDS

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**ABSTRACT:** Concerns with the harmful environmental impact of Portland cement manufacture on a global scale, has prompted an extensive search for clinker replacement materials and alternative low $CO_2$ cements. Amidst the development of radical new binder technologies there has been some resurgence in interest in Portland-cement's predecessor – hydraulic lime. This paper describes recent collaborative research, conducted by multidisciplinary engineering consultancy Ramboll and researchers at the BRE Centre for Innovative Construction Materials at the University of Bath, to develop modern, structural-grade, hydraulic-lime concretes. The paper details work to identify the pozzolanic additions, and combinations thereof, which result in structural strength concrete; and the results of a suite of tests to assess the structural and durability properties of the most-promising hydraulic-lime pozzolan concretes. The environmental credentials of hydraulic-lime pozzolan concretes in comparison with Portland cement-based concretes is then considered. In relation to these results the paper discusses the ways in which hydraulic-lime pozzolan concretes can be used, including case studies, and methods to adapt the mix proportions to optimise the properties for uses as varied as conservation of historic structures to engineered reinforced concrete construction. Finally, the paper discusses the future research that is needed to fully realise the potential of this new concrete.

**Keywords: Hydraulic lime, pozzolan, low-carbon concrete**

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## INTRODUCTION

Until recently lime-based materials have been understood to be weak and slow to set and have consequently been regarded as unsuitable for modern construction. The research described in this paper has, however, demonstrated the feasibility of producing modern, structural-grade concretes based on naturally hydraulic lime. Furthermore this research has demonstrated the potential for hydraulic lime-pozzolan concretes (HLPCs) as low-$CO_2$ alternatives to Portland cement (PC) concretes.

These innovative HLPCs should not be confused with 'Limecrete', or other commercially available lime-concretes, which are appropriate for the repair of historic buildings but not as alternatives to PC for structural applications.

### Historical context

Before the advent of PC, lime was the predominant binder for use in construction. Lime binders have a long and rich history and a lime-concrete floor slab discovered in Southern Israel in 1985 was dated back to 7000BC [1]. The practice of 'gauging' lime mortars and concretes with pozzolanic materials to improve performance is credited to the Greeks who used volcanic tuff with lime [2]. The Romans similarly utilized the volcanic ash from Pozzuoli, at the base of Mount Vesuvius. The technique then spread across Europe with the expansion of the Roman Empire, with lime-pozzolan concretes used to construct walls, floors, domes, bridges, aqueducts, harbours and cisterns [3]. By the middle ages the technology had been largely lost.

In the 19$^{th}$ century there was a revival in lime-pozzolan concrete technology and it was at this time that the first attempts to scientifically understand the properties of hydraulic-lime binders was undertaken by Smeaton (1724-92) and Vicat (1786-1861). Their work was followed by relatively rapid advances in binder technology and in 1824 Joseph Aspdin was granted the patent for a new artificial stone based on an improved binder he named Portland-cement. This 19$^{th}$ century 'proto-Portland cement' [2] was a forerunner of modern Portland-cements, which are now a mainstay of global human development. The development of PC meant that lime-pozzolans as a binder technology for concrete disappeared. Figure 1 depicts the three ages of lime-pozzolan concrete technology.

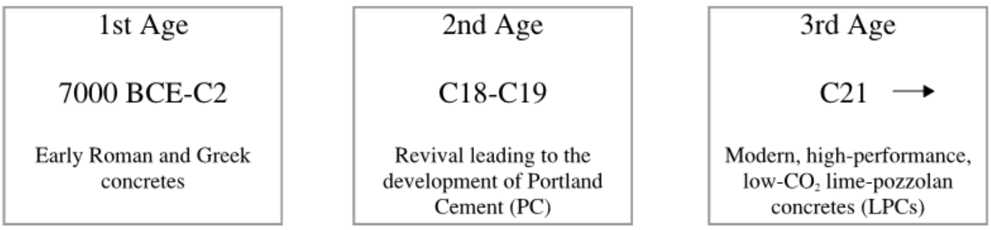


Figure 1 The three ages of lime-pozzolan concrete

Although this work was informed by historic practice and inspired by the durability of ancient concrete structures, these innovative HLPCs do not represent a return to a former technology as they utilise modern industrial waste ashes and exploit the advanced performance of the latest generation of admixtures.

**Initiation of the research**

In November 2008 Ramboll was contacted by an architectural practice interested in designing an eco-house with a doubly-curved concrete shell roof. The aspiration was to reduce the embodied impact of the structure by utilising a lime-based, as opposed to a PC-based, concrete. It was acknowledged that both the technical feasibility and the environmental desirability of this novel solution were contingent upon the structural capacity a concrete based on lime. The potential use of lime-concrete as an alternative to PC concrete for structural components was highlighted by Holmes and Wingate in 1997, but they acknowledged 'the science has not yet been developed' [3].

Prior to this research programme the only significant modern research on HLPC was that undertaken at the University of Aveiro, Portugal between 2009 and 2011 where HLPCs were being shown to attain 28-day compressive cube strengths ($f_{c28}$) of 11 MPa, with 20% of the hydraulic lime replaced with a waste residue of expanded clay production [4]. Subsequently, Cachim et al. demonstrated a maximum $f_{c28}$ of 17 MPa could be attained with 20% of the hydraulic lime replaced with a metakaolin [5]. However, Cachim et al made no further attempts to improve strength as their principal interest was the sustainable use of local materials. Although compressive strengths of around 10-15 MPa were ostensibly attainable, it was anticipated that compressive strengths in excess of 30MPa were going to be necessary to make the proposed HLPC shell roof both a feasible and desirable structural solution.

## RESEARCH TO DATE

**Phase 1- Relative efficiency of binary and ternary lime-pozzolan binders**

In the absence of a definitive source of information on hydraulic lime-pozzolan (HLP) binders, the initial phase of testing was a systematic study of the compressive strength development of HLP mortars prepared with NHL5, conforming to BS EN 459-1 [6], and a range of aluminosilicate mineral additions. In total 22 different mortar types were prepared and tested. The aim of this preliminary laboratory research was to identify a small number of additions, and/or combinations thereof, with the potential to result in structural strength HLPCs when scaled up from mortars to concretes.

The results showed that a ternary combination of 50% natural hydraulic lime (NHL5), 25% silica fume (SF) and 25% ground granulated blastfurnace slag (GGBS) resulted in a mortar with an average $f_{c28}$ of 28 MPa, at a water-to-binder (w/b) ratio of 0.5 [7]. This was eight times the strength of an equivalent mortar prepared with NHL5 alone and broadly speaking comparable with that of a low-heat cementitious mortar.

The results of this study allowed the pozzolanic additions, and combinations thereof, to be ranked from low to high efficacy, when used in conjunction with NHL5. The ternary combination of NHL5, SF and GGBS was shown to result in the greatest overall pozzolanic efficacy ($PE(\%)_{28d}$), attaining a maximum value of 94%. The four most promising combinations of additions, resulting in the highest strength mortars, which were chosen to be scaled up to HLPCs in the subsequent phase of testing, were:

| | |
|---|---|
| 70% NHL5, 15% FA & 15%MK, | (i) |
| 50% NHL5, 25% SF & 25% GGBS, | (ii) |
| 70% NHL5 & 30%SF, | (iii) |
| 50% NHL5, 25% SF & 25% FA | (iv) |

Figure 2 shows the compressive strength development of HLP combinations (i) to (iv) in comparison to 100% NHL5 and that of a proprietary formulated lime mortar (NHL-PC).

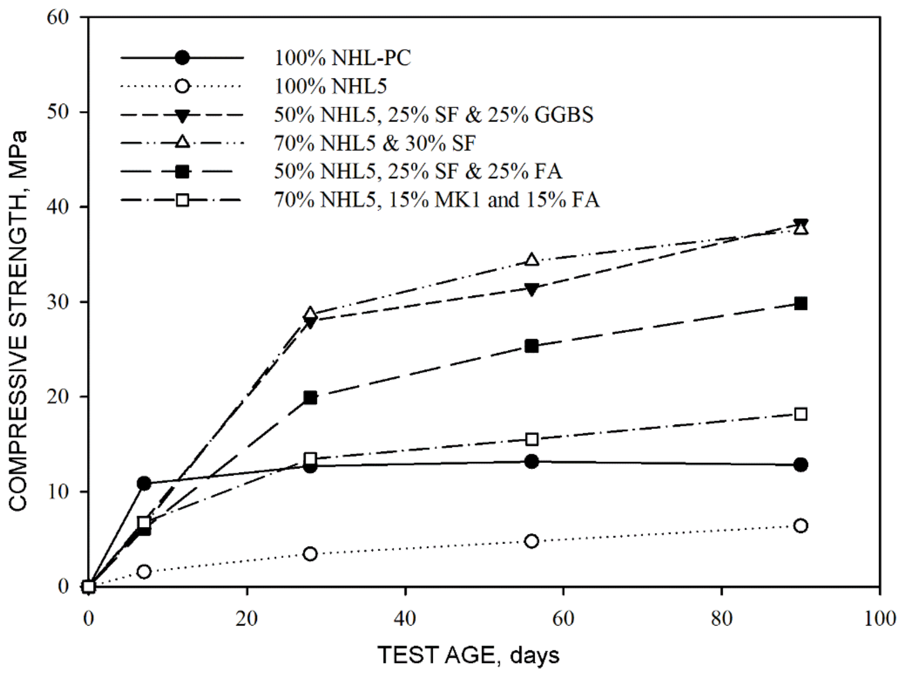


Figure 2 Compressive strength development of key mortars

**Phase 2 - Preliminary hydraulic lime-pozzolan concretes**

The aim of the second phase of testing was to investigate the structural and durability characteristics of four preliminary HLPCs. The compressive strength development, elastic modulus, linear shrinkage and rate of carbonation of the HLPCs were tested in comparison with Portland cement (PC) and blastfurnace cement (CIII/A) control concretes. Each concrete was prepared at three w/b ratios to investigate the effect of w/b ratio on the resultant properties of the hardened concretes. Additionally, the impact of curing conditions on compressive strength development was assessed by curing test specimens either in air (20 ± 0.5°C, 60-65% RH) or in a water bath (20 ± 0.5°C, 100% RH) [8].

The maximum $f_{c28}$ of the four HLPCs was 35MPa, attained by combining NHL5 (70% by mass) with SF (30% by mass) and curing the resultant concrete in water. The $f_{c28}$ of the equivalent air-cured concrete was 21MPa, 40% lower. Elastic modulus results demonstrated that the elasticity-compressive strength equation in Eurocode 2 for PC concretes, substantially overestimated the elastic modulus of the HLPCs.

The carbonation resistance of HLPCs was observed to be low in comparison to PC concretes. The results suggested that a HLPC incorporating 25% SF & 25% GGBS should provide sufficient protection for steel reinforcement for around 130 years. Increasing the depth of cover from 40 to 50mm increased this to over 200 years

The observed drying shrinkage of the HLPCs tested was, in the vast majority of cases, broadly in line with that of the PC-based control concretes over a 20 week period. SF was shown to be effective in minimising the impact of w/b ratio on the linear shrinkage of the resultant concretes. The HLPC comprising 70% NHL5, 15% FA & 15% MK was found to be highly sensitive to the variation in w/b ratio, raising concerns about the suitability of this ternary combination in practice.

As well as wanting to explore the mechanical characteristics of a range of HLPCs, a key aim of this industry-led research programme was to comment on the feasibility of constructing the lime-concrete dome that initiated the research. With $f_{c28}$ in excess of 30MPa having been shown feasible in the laboratory, the focus of the research shifted to converging on single HLPC appropriate for this project.

In this study the mechanical properties of the four alternative HLPCs were compared at a w/b ratio of 0.65. The greatest initial and long term strength gain, the highest strain at the maximum compressive strength ($\varepsilon_{c1}$), the greatest carbonation resistance and the least drying shrinkage, was exhibited by a HLPC comprising 50% NHL5, 25% SF and 25% GGBS [8]. As a result this ternary combination was selected for further investigation in Phase 3.

As well as successfully converging on a single ternary combination, this testing was effective in highlighting a number of practical considerations which were to inform the subsequent phase. For example, the results showed that the strongest and most durable HLPCs were produced at low w/b ratios. Having been unable to compact the HLPCs at low w/b ratios it was clear that a suitable superplasticiser (SP) needed to be identified going forward.

It was also recognised that the high dosage of SF used in Phases 1 & 2 of this study (25-30% by mass), might be untenable in future HLPCs due to commercial and legislative constraints. It was decided at this juncture to limit the use of SF to 10% of the total binder as is the case in PC based concretes [9].

**Phase 3 – An innovative concrete by design**

This phase of testing sought to address to fundamental question about this novel concrete technology. Firstly, could a HLPC be cast into structural elements with an appearance and surface finish similar to PC concrete? Secondly, could HLPC elements be designed to Eurocode 2 (EC2)?

The laboratory testing conducted during this phase comprised a sequence of experiments, aiming to explore and enhance the fresh behaviour and mechanical performance of a ternary combination of NHL5, GGBS and SF. Figure 3 illustrates the fresh behaviour of HLPC before and after the addition of a SP.

Figure 3 HLPC before and after the addition of SP

- By varying the mix proportions and experimenting with different proprietary SPs a HLPC with an $f_{c28}$ of 49MPa and an $f_{c90}$ excess of 60MPa was produced. This phase of testing culminated with the production and flexural testing of two reinforced HLPC beams. The failure load of the two beams was as predicted by EC2 (with due consideration of partial safety factors). The load-displacement behaviour of the two HLPC beams, in comparison to that of two identical PC beams is shown in Figure 4. Beams A_LP and A_ PC were under-reinforced and failed in a ductile manner whereas beams B_LP and B_PC were over-reinforced and failed suddenly when the compressive strength of the concrete was reached, as commonly observed in over-reinforced beams.

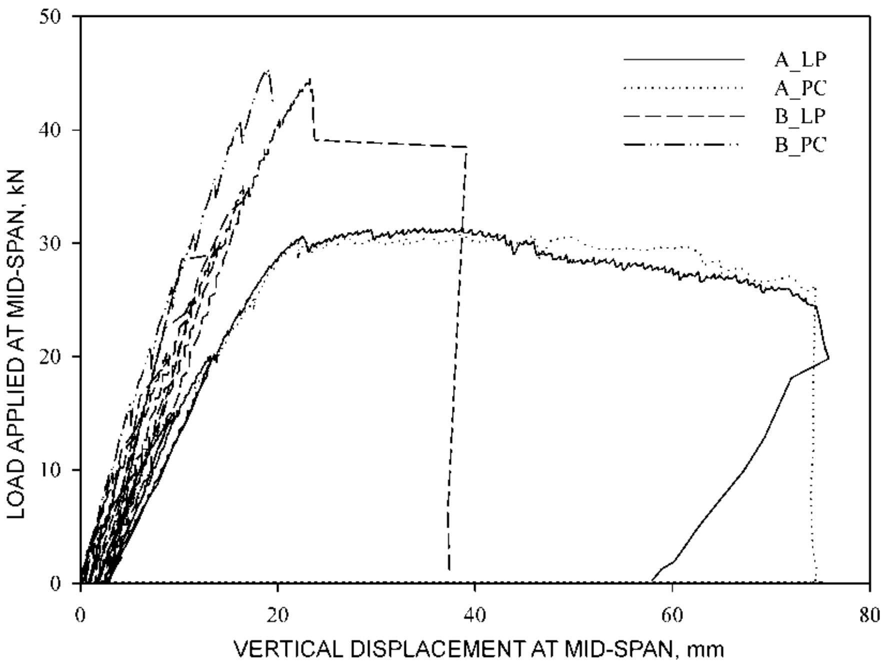


Figure 4 Load-displacement behaviour of under- and over-reinforced HLP and PC beams

**Phase 4 – So it can be done, but is it worth doing?**

The aim of this phase of the research was to assess whether these novel HLPCs were, or could be, desirable alternatives to PC concrete, particularly in the context of the industry wide search for low-$CO_2$ cements. Specifically, the embodied $CO_2$, embodied energy and binder intensity of a selection of these novel HLPCs were compared with PC concretes of the same $f_{c28}$. This included a laboratory study investigating the influence of the total binder content on the eco-efficiency of the resultant HLPCs.

This study demonstrated that the use of GGBS and SF in combination with NHL5 could realise savings in environmental impact, but that the potential savings were highly dependent on the boundaries of the analysis [10]. Specifically, the choice of allocation procedure was shown to have a profound effect on the selection of the 'greenest' binder when comparing PC and HLP concretes. Whereas in the case of GGBS it has been shown that economic allocation procedures maintain environmental benefits in comparison to PC [11], both mass and economic allocation procedures were shown to have a very detrimental effect on the environmental credentials of SF, see Figure 5.

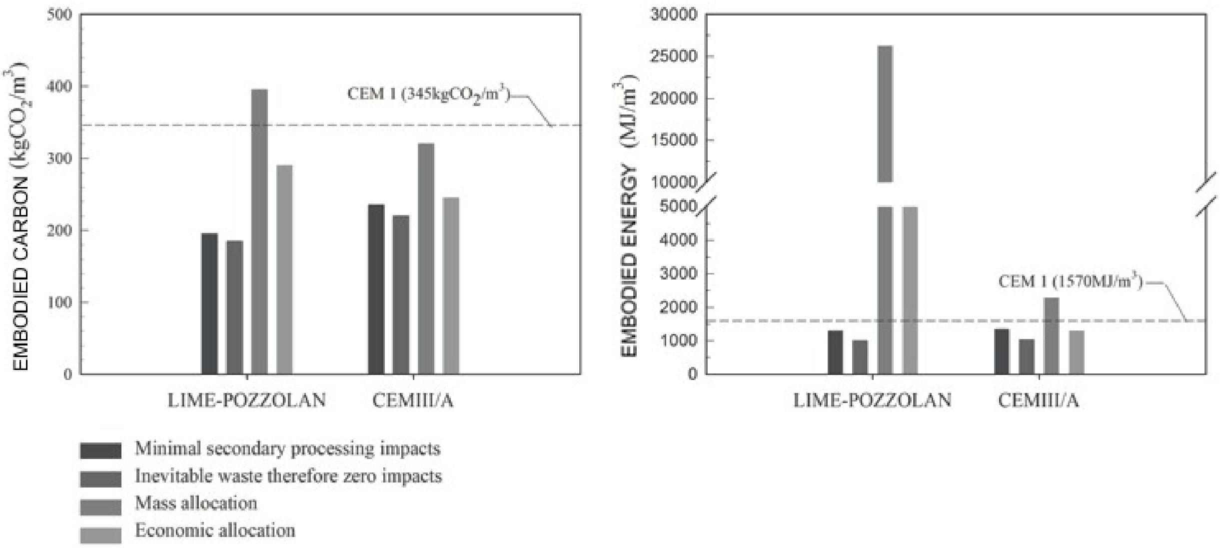


Figure 5 Influence of allocation methodology on the EC and EE of HLPC and CEMIII/A concretes

Assuming a 'waste' allocation and considering only the impacts associated with the secondary production of GGBS and SF, a HLPC with a binder comprising 50% NHL5, 40% GGBS & 10% SF and a $f_{c28}$ of 49MPa has an embodied $CO_2$ 43% less than a PC concrete of equivalent strength. The embodied $CO_2$ of this concrete is also 17% less than an equivalent strength CEMIII/A concrete. The embodied energy of this HLPC is 17-24% lower than that of the CEMI concrete (depending on inclusion of SP) and is 3-12% that of the CEMIII/A concrete.

## APPLICATION OF THIS TECHNOLOGY

In seeking to develop a HLPC suitable for reinforced structural elements, the research programme to date has tried to minimise differences between HLP and PC concretes. To maximise the impact of this material research and to exploit the potential environmental benefits of this binder technology, subsequent research will need to identify and amplify differential properties, which will open up the market for these alternative concretes. Whilst this research was initiated by a specific real-world project, for which a single structural-grade concrete was sought, the significance of innovation in binder technologies is the multifarious family of concretes that a new binder could bring about.

Potential early-adopter interest in this novel, low-$CO_2$ concrete technology created the opportunity to develop a second project-specific HLPC ‘product’ during the research programme. This real-world project was used as a case study for exploring the feasibility of a distinctly different application of this novel technology. In this case, keen to expose the innovative concrete in the building, the client sought a polished HLPC floor finish. This decorative screed incorporated oolitic limestone aggregate extracted from the site for which this flooring was developed. Although there is little recent precedence for polishing lime-concrete floors, examples of this technique, such as the decorative terrazzo floor at the Villa Saraceno, Italy laid in 1612, provided historical precedence for this solution [3].

This case study project was effective in addressing a number of questions about the possible application of this novel binder technology [12]. Development of the bespoke floor solution in conjunction with a specialist contractor demonstrated that HLPC can be diamond polished after two weeks, as per PC concrete and sealed with proprietary sealants. The embodied $CO_2$ of a 100mm thick polished HLPC floor was shown to be 45% less than a typical 100mm thick polished PC-concrete floor and 25% less than a conventional vinyl floor makeup. The embodied energy of a 100mm thick polished HLPC floor was 21% less than a typical 100mm thick polished PC-concrete floor and 37% less than a conventional vinyl floor makeup. The steel reinforcing mesh specified in the HLPC floor slab was shown to account for 43% of the embodied energy per $m^2$.

## DISCUSSION AND FUTURE RESEARCH DIRECTIONS

It is recognised that different applications and markets will require different material properties, leading to differentiation of the binder itself. For example in the conservation of historic buildings, lime-based materials are preferred as they exhibit a number of favourable characteristics that differentiate them from PC-based alternatives and make them preferable for the repair and maintenance of historic buildings, specifically breathability, permeability and flexibility. On this basis research is needed to understand the porosity, capillarity and elastic behaviour of HLPCs and importantly how these characteristics are affected by the nature and proportion of aluminosilicate additions in the binder. It is thought that a high proportion of SF in the binder may be detrimental in this respect. Similarly, lime-mortars are also advocated for autogenous-healing; the ability for free lime to carbonate in microcracks leading to self-healing mechanism. Whether or not HLPCs are also capable of being self-healing will depend on the long-term availability of free lime in the binder. These application-specific benefits might favour HLPCs with a low-pozzolanic content.

## Sustainability

In the 'green' building field the opposite may be true. Initial results suggest that the eco-efficiency of future HLPCs might be improved by increasing the proportion of pozzolanic materials in the binder (assuming the environmental impact of these materials is deemed lower than that of the NHL5 they replace). Testing has shown that a concrete prepared with a lower proportion of lime in the total binder (23% NHL5, 65% GGBS and 12% SF) has a $f_{c28}$ 3-4MPa greater than a concrete prepared with a higher proportion of lime in the total binder (53% NHL5, 35% GGBS and 12% SF), suggesting that the optimum dosage of aluminosilicate mineral additions, with respect to compressive strength, may exceed 77%. A substantial proportion of aluminosilicate mineral additions in the binder composition thus may prove beneficial from both an environmental and economic perspective.

## Durability

Lime-pozzolan concretes have a long history of use in marine structures; Pozzuoli Bay (Baianus Sinus), constructed in the first century BC, being just one example [13]. Although testing is still required to compare the performance of HLP and PC based concretes subject to aggressive exposure conditions, it is hypothesised that a high pozzolanic content may also be advantageous in HLPCs in aggressive environments, such as marine and sub-structural applications. Rapid Chloride Permeability Testing is needed to model the chloride ion diffusion coefficient of HLPCs. The high permeability of the HLPCs which are desirable for conservation and some other purposes can have a detrimental effect on some durability properties.

Other physical-chemical effects that warrant further performance testing and pre-qualification in the case of whether HLPCs are resistant to sulfate attack and alkali-silica reactions (ASR). The risk of the ettringite form of sulfate attack in HLPCs is thought to be low because of the low content of tricalcium aluminate ($C_3A$) in the binder and consequently the low amount of the AfM phase monosulfate in the concrete. However, it is acknowledged that there are several mechanisms of sulfate attack, including direct attack on $Ca(OH)_2$, which needs further investigation, before the sulfate resistance of HLPCs can be substantiated. The risk of ASR in HLPC is also thought to be low because NHL has a low sodium ($Na^+$) and potassium ($K^+$) ion content. Any $Na^+$ and $K^+$ present would be expected to react with the highly reactive pozzolanic materials, leaving no free $Na^+$ and $K^+$ in the system. Substantiation of the resistance of HLPCs to ASR would open up the possibility for HLPCs with a high recycled glass content.

## Alternative aluminosilicates

The combination of aluminosilicate additions that have been investigated in this research to date, may be currently deemed appropriate for the UK construction industry, but are unlikely to be the only, or the most appropriate, combination in other regional markets. There are indications that GGBS, FA and SF are becoming increasingly difficult to source in the UK [10]. Based on the results of laboratory testing of ternary HLPCs, there is substantial scope for broadening this field of enquiry into the testing and development of regional HLPCs that exploit locally available materials. Alternative mineral additions include industrial ashes, naturally occurring pozzolanic deposits, calcined clays and low-cost agro wastes.

Industrial waste ashes that may warrant further consideration in the development of future HLP cements include paper sludge ash [14], sewage sludge ash [15], municipal solid waste ash [16] and oil shale ash [17; 18]

Low-cost agro-wastes are also being investigated as partial PC replacement materials because of their pozzolanic properties. These materials might also warrant further investigation in the development of regional HLPCs: rice-husk ash [19], sugar cane bagasse [20], saw dust ash [21], corn cob ash [22], coconut husk ash [23], wheat straw ash [24], locust bean pod ash [25], palm oil fuel ash [26], cassava waste ash [27], olive waste ash [28] and periwinkle, oyster and snail shell ash [29]. Of this selection of waste ashes rice husk ash (RHA) is particularly interesting in the dialogue about future HLPCs, because of its wide scale availability and its high content of amorphous silica, which makes its oxide composition not dissimilar from that of SF.

### 'Cement' chemistry

The potential diversification of HLPC technology makes an in-depth study of the reaction chemistry and microstructure of HLP binders imperative. The insight gained from such an analysis will be hugely valuable in limiting the list of candidate pozzolanic mineral additions, and combinations thereof, and enable a design-led approach to the development of future HLPCs.

## FUTURE RESEARCH

In depth micro analysis is required to investigate the reaction kinetics and hydration products. Techniques identified as appropriate for such a study include isothermal conduction calorimetry, x-ray diffraction, thermal gravimetry and Fourier transform infrared spectrometry. Other techniques that might be valuable in studying the pore structure and phase assemblages of HLPCs include scanning electron microscopy, mercury intrusion porosimetry and x-ray computed tomography. General physiochemical results are needed, not only to explain the empirical results attained to date, but to refine and optimise future HLPCs.

### Choice of application

A design-led approach to the development of a range of tailored application- or region-specific concretes has the potential to improve the sustainability of the concrete industry. Such an approach puts emphasis on identifying specific requirements in-use and conducting testing at the front end. This might be expected to result in a shift focus, from generalised results to specialist testing and certification for use of HLPCs in different applications. For example, although this research has shown that HLPCs can be polished [12], the implementation of this material in floor systems will inevitably demand further testing to quantify the thermal performance of these novel concretes and their slip, stain, chemical and wear resistance in use.

## SUMMARY

This research programme was initiated by a real-world project aspiration and was required to converge on a single concrete, which represented a recommended and realisable solution to a specific design problem. The objective of the research presented in this paper was not, however, to discover a unique formula for a new marketable product, which might at some level compete with PC, but rather to push the boundaries of lime technology and where possible to create design space for future lime-based construction materials. 28-day compressive strengths in excess of 45MPa are boundary pushing, representing a step-change in our understanding of the potential strength, and rate of strength gain, associated with lime-based binders.

The results attained to date are recognised to be incomplete, with physiochemical analysis required to explain the phenomena observed laboratory. It is hoped that the research outlined in this paper, and detailed elsewhere, will inspire and inform a number of subsequent research projects and that the work to date might prove to be a platform for future research and development on HLPCs.

## ACKNOWLEDGEMENTS

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